Experimental report

15/02/2021

Proposal:	**EASY-758**	**Council:**	4/2020
Title:	In-situ measurement of the ionic liquids based electrolytes absorption within 1D Carbon NanoTube membrane		
Research area:	Soft condensed matter		

This proposal is a new proposal

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Samples: polystyrene, CNT, ionic liquid

Instrument	Requested days	Allocated days	From	To
NEXT	24	24	01/02/2021	02/02/2021

Abstract:

We recently found that ionic liquid based electrolytes confined in 1D carbon nanotube (CNT) membranes show a drastic increase of their ionic conductivity (thus the instant power of batteries). Compared to the same electrolytes in bulk, we indeed report conductivity gains by a factor up to 50 upon macroscopic 1D CNT confinement.

Up to now, we haven’t be able to characterize the IL absorption within the CNT membrane (homogeneous filling ? micro-porosity ? electrolyte within the polymer matrix ?). To use the CNT membranes as a battery separator we need to address this point. We propose here to measure in-situ the absorption of an IL based electrolytes within the CNT membrane and check its homogeneity using X-ray and Neutron imaging. First, we will acquire images of the empty membrane (deuterated polystyrene + empty CNT), then deposit a drop of a hydrogenated IL charged with CsTFSI salt (to maximise the neutron and X-ray absorption) and acquire images during 1h. We should be able to follow the absorption front while the IL fills the CNT by capillarity. Additionally, we plan to perform combined X-ray and neutron tomography to get information in 3D.

We apply for 1 day on NeXT.

In-situ measurement of the ionic liquids based electrolytes absorption within 1D Carbon NanoTube membrane

EASY-758

The goal of this experiment was to measure in-situ the absorption of an Ionic Liquids (IL) based electrolytes within a CNT membrane (Figure 1) using neutron imaging. We were in particular interested in the absorption kinetics of IL charged with lithium salt (electrolytes used for lithium batteries).

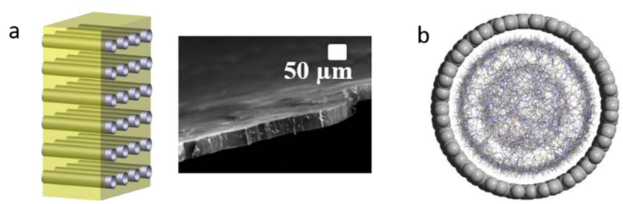

Figure 1. a) Schematic view of the 1D CNT membrane. Deuterated polystyrene (in yellow) has been used for the experiment. The CNT (in gray) are 4 nm in diameter and 100 µm long. SEM image of the CNT membrane. The porosity of the CNT membrane is ~ 6%. b) Organization of the IL molecules in concentric shell observed by MD simulations. [1]

Figure 2a shows a drop of an IL (OmimTFSI) on a 100 µm thick CNT membrane sitting onto a Teflon stand. The neat membrane has been counted for 100 seconds for reference. Then, an IL drop has been dropped onto the membrane (membrane is deutared and IL is hydrogenated). The square deep in the signal after 100 second (red arrow on Fig.2.b) corresponds to the shutdown of the beam necessary to the access to the hutch to perform this operation. When the beam is tuned back on, the time dependence of the intensity integrated over the red rectangle in Figure 2a shows a sharp decrease up to 2100 seconds. It is then followed by a smoother decrease. We attribute the first decrease in intensity to the IL filling the CNT pores. It seems that there is a two steps kinetics (even when plotted in log log). We need to measure for a longer time to interpret this second part of the signal.
We have demonstrated the feasibility of the experiment and will apply for beam-time.

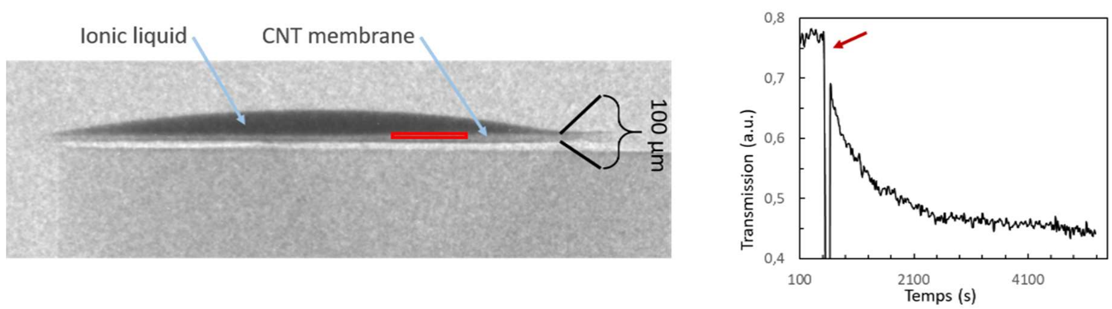

Figure 2. a) Image of a drop of IL on a CNT membrane. The red rectangle shows where the integration of the number of counts has been performed. b) Absorption profile. The red arrow shows when the IL is dropped.

[1] A. S. Pensado, F. Malberg, M. F. C. Gomes, A. A. H. Pádua, J. Fernández, and B. Kirchner, Interactions and Structure of Ionic Liquids on Graphene and Carbon Nanotubes Surfaces, RSC Adv. 4, 18017 (2014).